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Indian Standard

METHOD OF CHEMICAL ANALYSIS OF MISCH METAL

PART 5 DETERMINATION OF CARBON

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHOD OF CHEMICAL ANALYSIS OF MISCH METAL

PART 5 DETERMINATION OF CARBON

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METHOD OF CHEMICAL ANALYSIS OF MISCH METAL

PART 5 DETERMINATION OF CARBON

0. FOREWORD

0.1 This Indian Standard (Part 5) was adopted by the Indian Standards Institution on 16 January 1987, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Misch metal is an alloy of rare earth metals and is used for improving strength, fluidity and other properties of cast iron, steel, aluminium, magnesium and nickel alloys to which it is added. Chemical analysis of misch metal is covered in parts. In this part,a method for determination of carbon has been covered. Other parts are:

Part 1 Determination of cerium

Part 2 Determination of rare earths

Part 3 Determination of iron

Part 4 Determination of aluminium

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard (Part 5) covers the method for determination of carbon in various grades of misch metal as specified in IS : 4182-1967†

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070-1977‡) shall be employed in the test.

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*Rules for rounding off numerical values (*revised*).

†Specification for misch metal.

‡Specification for water for general laboratory use (*second revision*).

3. DETERMINATION OF CARBON BY DIRECT COMBUSTION METHOD

3.1 Outline of the Method — Carbon is oxidized to carbon dioxide by combustion in an atmosphere of oxygen and absorbed by ascarite. From the gain in mass of ascarite, the carbon content is calculated. As low as 0·05 percent carbon may be determined.

3.2 Apparatus and Reagents

3.2.1 *Apparatus* — As shown in Fig. 1.

3.2.1.1 *Combustion apparatus* — A mullite combustion tube heated in an electric furnace *E* heated by glow-bar going up to a temperature of 1 100°C in stages.

3.2.1.2 *Boats F* — Aluminium boats are heated to 1 000°C and allowed to cool in a desiccator before use.

3.2.1.3 *Oxygen purification system* — The oxygen gas first passes through concentrated sulphuric acid (*B*) and then through carbon dioxide absorbing tower (*C*) consisting of soda asbestos at the bottom and anhydrone at the top.

3.2.1.4 *Absorbing tower* — The tower (*L*) is filled with ascarite at the bottom and with anhydrone at the top.

3.2.1.5 *U-tubes* — Two U-tubes, one filled with zinc shots (*G*), and another with anhydrone (*K*).

3.2.1.6 *Traps* — One is filled with concentrated sulphuric acid saturated with chromic acid (*H*) and the other with concentrated sulphuric acid (J).

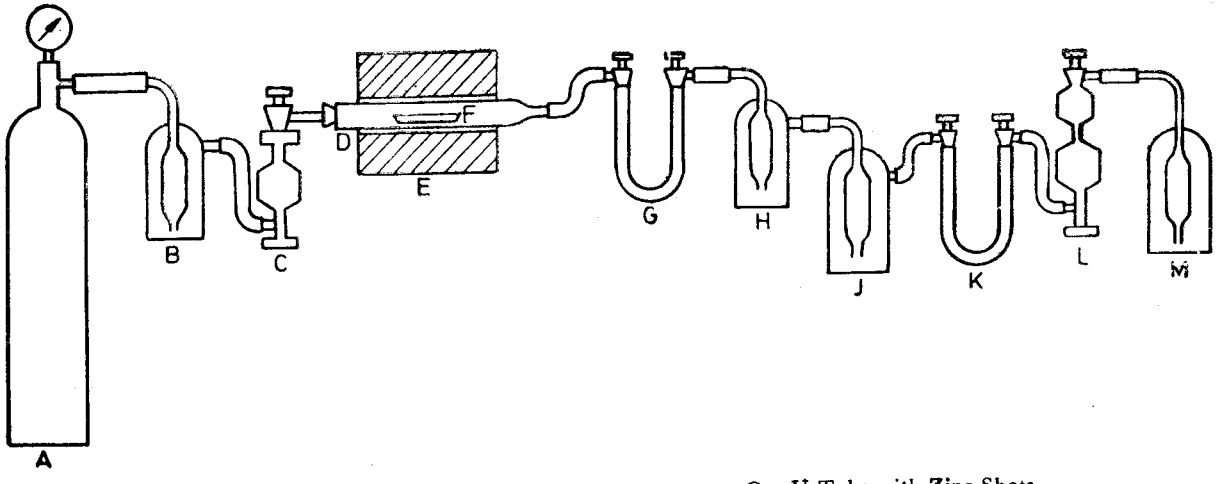
3.3 Procedure

3.3.1 Take about 5 g of sample in a beaker, wash with redistilled water thrice and then with analytical grade acetone thrice to remove any organic impurities on the surface. Keep sample in an oven for about 3-4 hours at 110°C.

3.3.2 Meanwhile switch on the furnace and raise the temperature slowly to 1 100°C and maintain at this temperature for an hour.

3.3.3 Open the stop cocks on *C, G, K* and *L* first. Then open the cylinder carefully to allow oxygen to pass through the apparatus at a rate of about 30 ml per minute (*see* Note).

NOTE — Ensure that there is no back suction during oxidation.



A = Oxygen Cylinder
B = H_2SO_4 Trap
C = CO_2 Absorbing Tower
D = Combustion Tube
E = Furnace
F = Boat
G = U-Tube with Zinc Shots
H = Chromic Acid Trap
J = H_2SO_4 Trap
K = U-Tube with Anhydrone
L = CO_2 Absorbing Tower
M = H_2SO_4 Trap

FIG. 1 APPARATUS FOR DETERMINATION OF CARBON

3.3.4 After half an hour, disconnect the tower *L* closing the stop cock at the U-tube (*K*) first and then the stop cock at the top of tower (*L*). Clean the outside surface of tower with tissue paper and weigh (M_1).

3.3.5 Connect the tower (*L*) back. Introduce the boat (**3.2.1.2**) into the hot zone of the mullite tube by opening at its mouth, in the minimum time possible (less than half a minute). Avoid touching the boat with hands. Connect back the oxygen flow through the mullite tube. Allow the gas to flow under these conditions for 30 minutes.

3.3.6 Disconnect the tower *L* and weigh (M_2) as in **3.3.4**. The blank ($M_2 - M_1$) should be less than 1·0 mg.

3.3.7 Meanwhile remove the boat from the furnace and connect back the oxygen flow.

3.3.8 Weigh 2·500 g sample and spread it in another boat.

3.3.9 Connect the tower (*L*) back and then introduce the boat with sample into the hot zone of the combustion tube, close the tube and pass oxygen gas for half an hour as in **3.3.5**. Watch the tower *H* or *J*. A lower pressure in the furnace side indicates rapid consumption of oxygen. If this is leading to the entrance of concentrated sulphuric acid into U-tube (*G*), remove the stopper on *G* and proceed as in **3.3.11** and **3.3.12**.

3.3.10 Disconnect the tower (*L*) and weigh (M_3) as in **3.3.4**. Whenever, this tower is not connected in the apparatus, it is kept in a desiccator. $M_3 - M_2$ gives the mass of CO_2 liberated.

3.3.11 Remove the boat from the furnace and ensure for the complete combustion of the sample.

3.3.12 Switch off the furnace. Close the oxygen flow from the cylinder. Close stop cocks on *C*, *G* and *K*.

3.4 Calculation

$$\text{Carbon, percent} = [(M_3 - M_2) - (M_2 - M_1)] \times 10{\cdot}91$$

where

$M_3 - M_2$ = mass of carbondioxide found, and

$M_2 - M_1$ = mass of carbondioxide found in blank.

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